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Direct Measurement of ¹⁴CO₂ by Liquid Scintillation Counting

The problem:

To develop a technique for the direct measurement of carbon-14 in carbon dioxide. Previous methods suffered from such drawbacks as nonquantitative absorption or reaction of the CO₂, quenching from the addition of absorbents or reactants, and isotope effects during successive reactions.

The solution:

A liquid scintillation counting technique for measurement of ¹⁴C activity. The carbonaceous sample is first converted into CO₂, and the ¹⁴CO₂ is then measured by a liquid scintillation counter. This method has high counting efficiency and eliminates many of the basic problems encountered with previous measuring techniques.

How it's done:

Three different counting systems have been used with equal success: a room temperature single-multiplier phototube (RCA-8575) system; $a-20^{\circ}$ C single-multiplier phototube (DuMont-6292) system; and a 0° C coincidence liquid scintillation counter.

Two scintillator solutions were used to match emission from the solution with response of the multiplier phototubes. With the RCA-8575 multiplier phototube, a solution of 3 g of PPO in a liter of toluene was used; with the DuMont-6292 multiplier phototube, a solution of 7 g of PPO and 0.5 g of M₂-POPOP in a liter of toluene was used.

The CO₂ gas samples were prepared by normal vacuum line manipulations. The CO₂ was condensed with liquid nitrogen onto a previously degassed and frozen volume of the scintillator solution. The sample tubes were then sealed by flame, minimizing the volume of space above the scintillator solution.

The solubility of CO_2 in toluene base scintillator solution is sufficient to allow the ¹⁴C measurement at room temperature. By the use of a special type of sample container, the space above the solution was minimized to the extent that greater than 99% of the CO_2 was dissolved in the solution.

To determine the concentration quenching of CO_2 , samples were prepared with varying concentrations of CO_2 . The relative scintillation yields were determined by the Compton edge technique. There was no detectable quenching up to 5 cc (STP) per ml of liquid scintillator solution.

To determine the counting efficiency, several samples of known volume of 14 C containing CO₂ were dissolved in 2 ml of scintillator solution and counted in a room temperature single-multiplier phototube system. The counting rates were determined by the integral counting technique. Aliquots of the same gas were counted in a gas counter. The extrapolated integral counting rates of samples in the liquid scintillators agreed with $\pm 2\%$ of the absolute disintegration rate obtained with the gas counter.

This method can be useful for either assay or absolute measurement of ^{14}C radioactivities in samples converted into CO₂. Counting efficiencies of greater than 90% are obtained with low backgrounds. With an integral counting technique, a counting efficiency of $(100 \pm 2)\%$ was obtained. The lower level of detection of ^{14}C by this method is 10^{-7}C i per cc (STP) of CO₂ for a 10 cc sample (about 2 disintegrations per minute).

Notes:

1. The use of scintillation counters is not new, but this particular measurement is a novel application.

(continued overleaf)

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- 2. The technique can be used to achieve a percent substitution reaction, and may be of interest to university and hospital laboratories as an analytical technique.
- 3. Inquiries concerning this report may be directed to:

Office of Industrial Cooperation Argonne National Laboratory 9700 South Cass Avenue Argonne, Illinois 60439 Reference: B69-10092

> Source: D. L. Horrocks Chemistry Division (ARG-10237)

Patent status:

Inquiries about obtaining rights for commercial use of this innovation may be made to:

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